



US007470824B2

(12) **United States Patent**
Tanaka et al.(10) **Patent No.:** **US 7,470,824 B2**
(45) **Date of Patent:** **Dec. 30, 2008**(54) **ADAMANTANE DERIVATIVE AND PROCESS FOR PRODUCING THE SAME**(75) Inventors: **Shinji Tanaka**, Chiba (JP); **Hidetoshi Ono**, Chiba (JP); **Kouichi Kodoi**, Chiba (JP); **Naoyoshi Hatakeyama**, Chiba (JP)(73) Assignee: **Idemitsu Kosan Co., Ltd.**, Tokyo (JP)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 400 days.

(21) Appl. No.: **10/540,547**(22) PCT Filed: **Dec. 18, 2003**(86) PCT No.: **PCT/JP03/16258**§ 371 (c)(1),
(2), (4) Date: **Dec. 13, 2005**(87) PCT Pub. No.: **WO2005/058675**PCT Pub. Date: **Jul. 15, 2004**(65) **Prior Publication Data**

US 2006/0149073 A1 Jul. 6, 2006

(30) **Foreign Application Priority Data**

Dec. 25, 2002 (JP) 2002-374659

(51) **Int. Cl.**
C07C 43/18 (2006.01)(52) **U.S. Cl.** **568/665**(58) **Field of Classification Search** **568/665**
See application file for complete search history.(56) **References Cited****U.S. PATENT DOCUMENTS**6,440,636 B1 8/2002 Ushirogouchi et al.
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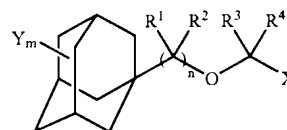
Primary Examiner—Rei-tsang Shiao

Assistant Examiner—Joseph R Kosack

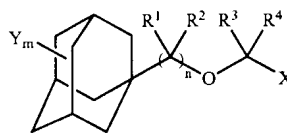
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(57) **ABSTRACT**

Provided is an adamantane derivative represented by Formula (I) or (II):



(I)



(II)

wherein X represents a halogen atom; Y represents an alkyl group having 1 to 10 carbon atoms, a halogenated alkyl group having 1 to 10 carbon atoms, a halogen atom or a hetero atom-containing group; R¹ to R⁴ represent independently hydrogen, a halogen atom, an alkyl group having 1 to 10 carbon atoms or a halogenated alkyl group having 1 to 10 carbon atoms; m represents an integer of 0 to 15, and n represents an integer of 0 to 10; and excluded is a case where in Formula (I), m and n are 0 at the same time and R³ and R⁴ are a hydrogen atom at the same time.

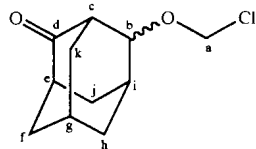
Capable of being provided is a novel adamantane derivative which is useful as a modifying agent for a resin for a photoresist and a dry etching resistance-improving agent in the photolithography field, agricultural and medical intermediates and other various industrial products.

3 Claims, No Drawings

11

Example 2

Synthesis of (chloromethyl)
(4-oxo-2-adamantyl)Ether Represented by a
Structural Formula



A separable flask having a volume of one liter equipped with a nozzle for introducing hydrogen chloride gas was equipped with a stirring device and charged with 50.0 g (301 mmol) of 4-oxo-2-adamantanol, 13.6 g (450 mmol) of paraformaldehyde, 36.2 g (300 mmol) of magnesium sulfate and 650 ml of dried dichloromethane, and it was cooled to 0° C. on an ice bath and stirred. Hydrogen chloride gas generated by mixing 300.7 g (5 mole) of sodium chloride with 700 ml of conc. sulfuric acid was blown therein through the nozzle for one hour. After further stirring for 3 hours, magnesium sulfate was filtered, and then the solution was analyzed by gas chromatography to confirm that 4-oxo-2-adamantanol was completely converted and that the intended product was obtained at a selectivity of 93.2%. Hydrogen chloride and dichloromethane were removed, and then refining was carried out by distillation to isolate 55.0 g (256 mmol, yield 85.2%, GC purity 98.8%) of the intended product.

The analytical results of the above compound are shown below.

Nuclear magnetic resonance spectrometry (NMR): CDCl_3 , $^1\text{H-NMR}$ (500 MHz): 1.66 to 1.69 (m), 1.75 to 1.78 (m), 1.89 to 2.12 (m), 2.20 (m), 2.25 (m), 2.82 (m), 2.40 (dq, $J=13.0$ Hz, 2.8 Hz), 2.51 (s), 2.54 (s), 2.79 (s), 3.94 (t, $J=3.5$ Hz, 1H, b^2), 4.31 (q, $J=2.7$ Hz, 1H, b^1), 5.52 (s, 2H, a^1), 5.55 (dd, $J=5.4$ Hz, 17.6 Hz, 2H, a^2), $^{13}\text{C-NMR}$ (127 MHz): 26.26 (g^1), 26.39 (g^2), 29.98 (f^2 or h^2 or j^2 or k^2), 30.95 (i^2), 31.26 (i^1), 32.46 (f^2 or h^2 or j^2 or k^2), 32.98 (f^1 or h^1 or j^1 or k^1), 33.44 (f^2 or h^2 or j^2 or k^2), 34.99 (f^1 or h^1 or j^1 or k^1), 37.80 (f^1 or h^1 or j^1 or k^1), 38.68 (f^2 or h^2 or j^2 or k^2), 38.78 (f^1 or h^1 or j^1 or k^1), 45.31 (e^2), 46.18 (e^1), 50.52 (c^1), 51.07 (c^2), 79.69 (b^1), 79.82 (a^2), 80.44 (b^2), 83.97 (a^1), 213.86 (d^2), 214.96 (d^1) (superscript numeral 1 represents a principal isomer, and superscript numeral 2 represents a subsidiary isomer)

Gas chromatography mass spectrometry (GC-MS): EI 216 ($M^+ + 2$, 2.9%), 214 (M^+ , 8.7%), 148 (29.2%), 79 (100%)

Boiling point: 160 to 161° C./0.2 kPa

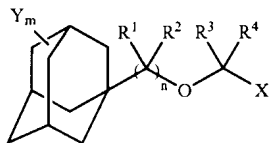
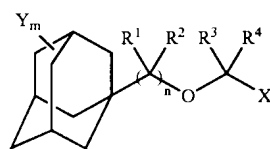
INDUSTRIAL APPLICABILITY

The adamantane derivatives of the present invention are novel (monohalogen-substituted methyl) (adamantyl group-containing alkyl)ethers, and they are useful as a modifying agent for a resin for a photoresist and a dry etching resistance-improving agent in the photolithography field, agricultural and medical intermediates and other various industrial products.

12

What is claimed is:

1. A substituted adamantane of Formula (I) or (II):



wherein

X represents a halogen atom;

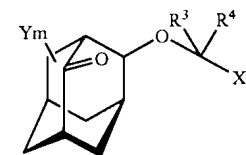
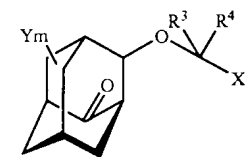
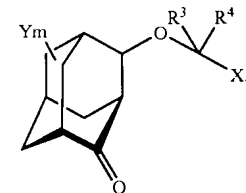
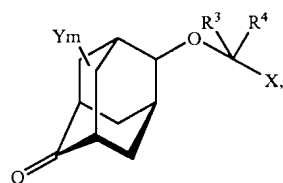
Y is a C_1 - C_{10} alkyl group, a halogenated C_1 - C_{10} alkyl group, a halogen atom or a hetero atom-containing group;

R^1 and R^2 represents, independently, hydrogen, a halogen atom, a C_1 - C_{10} alkyl group or a halogenated C_1 - C_{10} alkyl group;

R^3 and R^4 represent, independently, hydrogen, a C_1 - C_{10} alkyl group, or a C_1 - C_{10} halogenated alkyl group, wherein R^3 and R^4 cannot both be hydrogen in Formula (I); wherein, in the formula (I), m represents an integer of 0 to 15, and n represents an integer of 1 to 10;

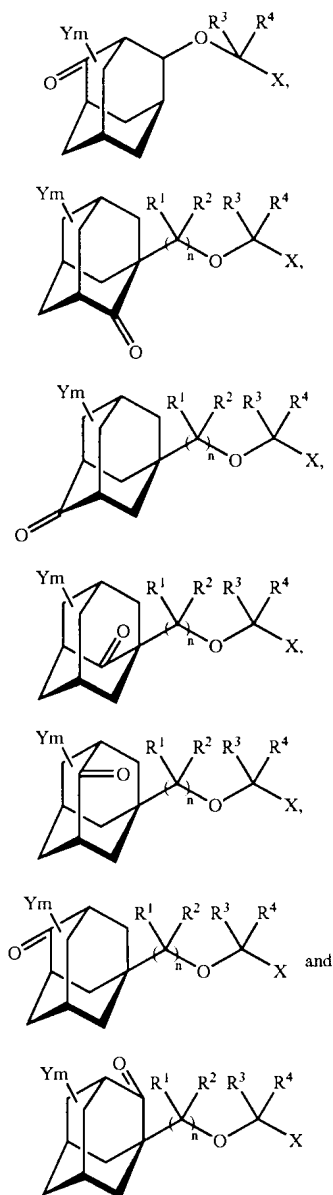
wherein, in the Formula (II), m represents an integer of 1 to 15.

2. A substituted adamantane selected from the group consisting of



13

-continued



14

wherein

X represents a halogen atom;

(Ile) Y is a C₁-C₁₀ alkyl group, a halogenated C₁-C₁₀ alkyl group, a halogen atom or a hetero atom-containing group;

5 R¹ and R² represent, independently, hydrogen, a halogen atom, a C₁-C₁₀ alkyl group or a halogenated C₁-C₁₀ alkyl group;

(Ia) 10 R³ and R⁴ represent, independently, hydrogen, a C₁-C₁₀ alkyl group, or a C₁-C₁₀ halogenated alkyl group, wherein R³ and R⁴ cannot both be hydrogen in Formulae (Ia-Ie);

wherein, in the formulae (Ia-Ie), m represents an integer of 0 to 13, and n represents an integer of 1 to 10;

15 wherein, in the Formula (IIa-IIe), m represents an integer of 1 to 13.

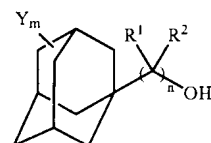
(Ib) 3. A process for producing the substituted adamantane of claim 1, comprising

20 reacting an alcohol comprising an adamantyl group represented by Formula (III) or (IV):

(III)

(Ic)

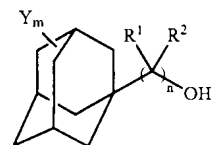
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(IV)

(Id)

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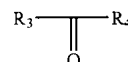
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(Ie)

wherein in Formula (III), n represents an integer of 1 to 10; with a carbonyl compound represented by Formula (V):

(V)

40



(If)

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and at least one hydrogen halide gas; wherein R³ and R⁴ in Formula (V) are not both hydrogen when the carbonyl compound of formula (V) is reacted with the adamantyl group of Formula (III), and wherein m represents an integer of 1 to 13.

* * * * *

DOCKET NO: 273504US0PCT

IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF :
SHINJI TANAKA, ET AL. : EXAMINER: J. R. KOSACK
SERIAL NO: 10/540,547 :
FILED: DECEMBER 13, 2005 : GROUP ART UNIT: 1626
FOR: ADAMANTANE DERIVATIVE :
AND PROCESS FOR PRODUCING THE
SAME

AMENDMENT AND REQUEST FOR RECONSIDERATION

COMMISSIONER FOR PATENTS
ALEXANDRIA, VIRGINIA 22313

SIR:

In response to the Office Action dated March 19, 2008, please amend the above-identified application as follows:

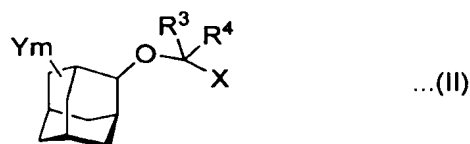
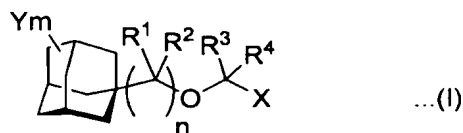
Amendments to the Claims are reflected in the listing of claims which begins on page 2 of this paper.

Remarks/Arguments begin on page 6 of this paper.

IN THE CLAIMS

Please amend the claims as follows:

Claim 1 (Currently Amended): ~~[[An]]~~ A substituted adamantane derivative
~~characterized by having a structure represented by of~~ Formula (I) or (II):



wherein

X represents a halogen atom;

~~Y represents an~~ is a C₁-C₁₀ alkyl group having 1 to 10 carbon atoms, a halogenated C₁-C₁₀ alkyl group ~~having 1 to 10 carbon atoms~~, a halogen atom or a hetero atom-containing group;

R¹ ~~[[to R⁴]]~~ and R² represent, independently, hydrogen, a halogen atom, ~~[[an]]~~ a C₁-C₁₀ alkyl group having 1 to 10 carbon atoms or a halogenated C₁-C₁₀ alkyl group ~~having 1 to 10 carbon atoms~~;

R³ and R⁴ represent, independently, hydrogen, a C₁-C₁₀ alkyl group, or a C₁-C₁₀ halogenated alkyl group.

wherein R³ and R⁴ cannot both be hydrogen in Formula (I);

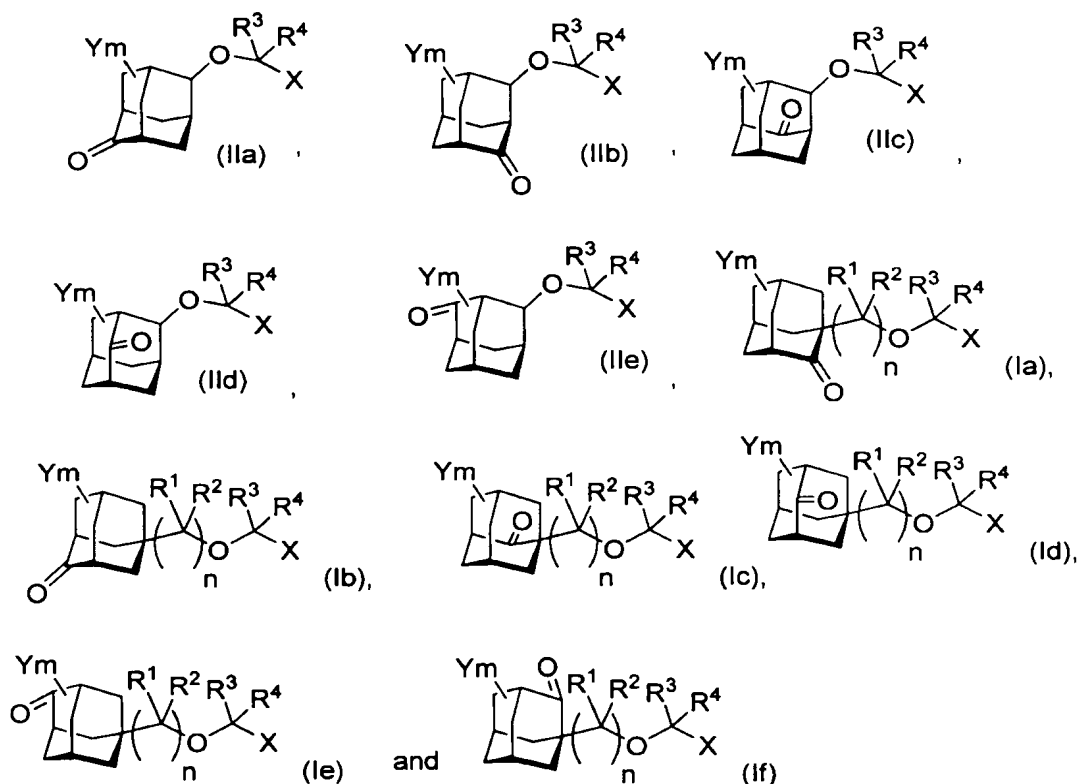
wherein, in the formula (I), m represents an integer of 0 to 15, and

n represents an integer of 1 to 10 0 to 10; and

wherein, in the Formula (II), m represents an integer of 1 to 15

~~excluded is a case where in Formula (I), m and n are 0 at the same time and R³ and R⁴ are a hydrogen atom at the same time.~~

Claim 2 (Currently Amended): [[The]] A substituted adamantane selected from the group consisting of



wherein

X represents a halogen atom;

Y is a C₁-C₁₀ alkyl group, a halogenated C₁-C₁₀ alkyl group, a halogen atom or a hetero atom-containing group;

R¹ and R² represent, independently, hydrogen, a halogen atom, a C₁-C₁₀ alkyl group or a halogenated C₁-C₁₀ alkyl group;

R³ and R⁴ represent, independently, hydrogen, a C₁-C₁₀ alkyl group, or a C₁-C₁₀ halogenated alkyl group,

wherein R³ and R⁴ cannot both be hydrogen in Formulae (Ia-If);

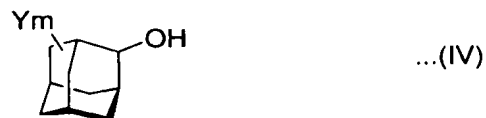
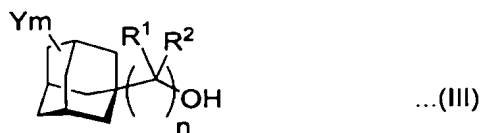
wherein, in the formulae (Ia-If), m represents an integer of 0 to 13, and
n represents an integer of 1 to 10 ~~0 to 10~~; and

wherein, in the Formula (IIa-IIe), m represents an integer of 1 to 13

derivative according to claim 1, wherein in Formula (I) or (II), Y represents =O
formed by allowing two Y's to be put together.

Claim 3 (Currently Amended): A process for producing the substituted adamantane derivative of claim 1, comprising

reacting an alcohol comprising an adamantyl group represented by Formula (III) or (IV):



wherein in Formula (III), n represents an integer of 1 to 10;

wherein X, Y, R¹, R², m and n are the same as described above, with a carbonyl
compound represented by Formula (V):



and at least one hydrogen halide gas; ~~to obtain the adamantane derivative; wherein R³~~
~~and R⁴ are the same as described above, and wherein when m and n are 0 at the same time in~~
~~Formula (III) described above, R³ and R⁴ in Formula (V) are not both hydrogen when the~~
~~carbonyl compound of formula (V) is reacted with the adamantyl group of Formula (III), and~~
~~wherein m represents an integer of 1 to 13 are not a hydrogen atom.~~

Claims 4-8 (Cancelled).

REMARKS/ARGUMENTS

Claims 4-8 are cancelled.

Support for each amended claim is found at the originally filed claims and throughout the specification.

Upon entry of the amendment, Claims 1-3 will be active.

No new matter is believed to have been added.

Any duplicate claim objection of Claim 8 is obviated by cancellation of Claim 8.

The enablement rejection of Claims 3-8 is respectfully traversed. The rejection of Claims 4-8 is obviated by cancellation of these claims. Present Claim 1, and by extension present dependent Claim 3, has been amended to contain the features “R³ and R⁴ represent, independently, hydrogen, a C₁-C₁₀ alkyl group, or a C₁-C₁₀ alkyl group...” Because Claims 1 and 3 do not describe that R³ and / or R⁴ can be a halogen, Claims 1 and 3 are believed to be definite. Withdrawal of enablement rejection is respectfully requested.

The indefiniteness rejection of Claims 2, 5 and 7 is respectfully traversed. The rejection of Claims 5 and 7 is obviated by cancellation of these claims. Claim 2 has been amended such that the term “by allowing two Y’s to be put together” has been removed from Claim 2. Accordingly, it is believed that Claim 2 is definite. Withdrawal of the rejection is respectfully requested.

The indefiniteness rejection of Claims 1-8 for containing features such as “having 1 to 10 carbon atoms” is respectfully traversed. The rejection of Claims 4-8 is obviated by cancellation of these claims. Applicants respectfully submit that one of ordinary skill in the art would readily understand what the phrase “having 1 to 10 carbon atoms” means, and therefore that the term is definite. Nevertheless, as a courtesy to the Office, Applicants have

replaced the phrase "having 1 to 10 carbon atoms" with the phrase "C₁-C₁₀." Claims 1-3 are definite. Withdrawal of the rejection is requested.

The anticipation rejection of Claims 1-8 as being unpatentable in view of Ben-David is respectfully traversed. The rejection of Claims 4-8 is obviated by cancellation of these claims. Present Claim 2 comprises oxadamantyl moieties. These moieties are not described or suggested by Ben-David. Further, present Claims 1 and 3 do not encompass the compound of Ben-David cited at page 6 of the Official Action. Ben-David therefore cannot anticipate present Claims 1-3. Withdrawal of the anticipation rejection is requested.

The obviousness rejection of Claims 4, 6 and 8 as being unpatentable in view of Okada is obviated by cancellation of these claims.

Applicants submit the present application is now in condition for allowance. Early notification to this effect is earnestly solicited.

Respectfully submitted,

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MAIER & NEUSTADT, P.C.
Norman F. Oblon



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(OSMMN 08/07)

10/540,547	ADAMANTANE DERIVATIVE AND PROCESS FOR PRODUCING THE SAME	08-20-2009::18:20:19
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bibliographic Data

Application Number:	10/540,547	Customer Number:	22850
Filing or 371 (c) Date:	12-13-2005	Status:	Patented Case
Application Type:	Utility	Status Date:	12-10-2008
Examiner Name:	KOSACK, JOSEPH R	Location:	ELECTRONIC
Group Art Unit:	1626	Location Date:	-
Confirmation Number:	7194	Earliest Publication No:	US 2006-0149073 A1
Attorney Docket Number:	273504US0PCT	Earliest Publication Date:	07-06-2006
Class / Subclass:	568/665	Patent Number:	7,470,824
First Named Inventor:	Shinji Tanaka , Chiba, (JP)	Issue Date of Patent:	12-30-2008

Title of Invention:	ADAMANTANE DERIVATIVE AND PROCESS FOR PRODUCING THE SAME
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ADAMANTANE DERIVATIVE AND PROCESS FOR PRODUCING THE SAME

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his application is officially maintained in electronic form. To View: Click the desired Document description. To Download and Print: Check the desired document(s) and click PDF.

Bibliographic Data

File Room Date	Document Code	Document Description	Document Category	Page Count
2-17-2009	COCOUT	Certificate of Correction - Post Issue Communication	PROSECUTION	1
1-14-2009	COCIN	Request for Certificate of Correction	PROSECUTION	4
1-14-2009	N417	EFS Acknowledgment Receipt	PROSECUTION	2
1-14-2009	LET.	Miscellaneous Incoming Letter	PROSECUTION	1
2-11-2008	OA.EMAIL	Email Notification	PROSECUTION	1
2-10-2008	ISSUE.NTF	Issue Notification	PROSECUTION	1
1-26-2008	IFEE	Issue Fee Payment (PTO-85B)	PROSECUTION	1
1-26-2008	WFEE	Fee Worksheet (PTO-875)	PROSECUTION	2
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3-17-2008	1449	List of References cited by applicant and considered by examiner	PRIOR ART	1
3-03-2008	NOA	Notice of Allowance and Fees Due (PTOL-85)	PROSECUTION	7
3-03-2008	SRNT	Examiner's search strategy and results	PROSECUTION	1
3-03-2008	SRFW	Search information including classification, databases and other search related notes	PROSECUTION	1
3-03-2008	IIFW	Issue Information including classification, examiner, name, claim, renumbering, etc.	PROSECUTION	1
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3-03-2008	BIB	Bibliographic Data Sheet	PROSECUTION	1
3-03-2008	OA.EMAIL	Email Notification	PROSECUTION	1
5-27-2008	A...	Amendment/Req. Reconsideration-After Non-Final Reject	PROSECUTION	1
5-27-2008	CLM	Claims	PROSECUTION	4
5-27-2008	REM	Applicant Arguments/Remarks Made in an Amendment	PROSECUTION	2
5-27-2008	N417	EFS Acknowledgment Receipt	PROSECUTION	2
5-27-2008	LET.	Miscellaneous Incoming Letter	PROSECUTION	1
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3-19-2008	FWCLM	Index of Claims	PROSECUTION	1
3-19-2008	SRFW	Search information including classification, databases and other search related notes	PROSECUTION	1
3-19-2008	BIB	Bibliographic Data Sheet	PROSECUTION	1
3-19-2008	1449	List of References cited by applicant and considered by examiner	PRIOR ART	1
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3-19-2008	SRNT	Examiner's search strategy and results	PROSECUTION	355
3-19-2008	NPL	NPL Documents	PROSECUTION	5
3-19-2008	OA.EMAIL	Email Notification	PROSECUTION	1
3-30-2006	IDS	Information Disclosure Statement (IDS) Filed (SB/08)	PROSECUTION	3
3-30-2006	FOR	Foreign Reference	PRIOR ART	137
3-30-2006	FOR	Foreign Reference	PRIOR ART	2
3-30-2006	NPL	NPL Documents	PRIOR ART	3
3-30-2006	NPL	NPL Documents	PRIOR ART	1
3-30-2006	NPL	NPL Documents	PRIOR ART	5
3-30-2006	NPL	NPL Documents	PRIOR ART	3
3-28-2006	M903	Notice of DO/EO Acceptance Mailed	PROSECUTION	2

1-13-2005	LET.	Miscellaneous Incoming Letter	PROSECUTION	3
1-13-2005	OATH	Oath or Declaration filed	PROSECUTION	4
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1-23-2005	M905	Notice of DO/EO Missing Requirements Mailed	PROSECUTION	2
1-24-2005	TRNA	Transmittal of New Application	PROSECUTION	3
1-24-2005	136A	Authorization for Extension of Time all replies	PROSECUTION	3
1-24-2005	SPEC	Specification	PROSECUTION	25
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1-24-2005	REM	Applicant Arguments/Remarks Made in an Amendment	PROSECUTION	1
1-24-2005	ADS	Application Data Sheet	PROSECUTION	3
1-24-2005	IDS	Information Disclosure Statement (IDS) Filed (SB/08)	PROSECUTION	2
1-24-2005	NPL	NPL Documents	PRIOR ART	5
1-24-2005	NPL	NPL Documents	PRIOR ART	6
1-24-2005	NPL	NPL Documents	PRIOR ART	3
1-24-2005	NPL	NPL Documents	PRIOR ART	4
1-24-2005	NPL	NPL Documents	PRIOR ART	7
1-24-2005	NPL	NPL Documents	PRIOR ART	2
1-24-2005	FRPR	Certified Copy of Foreign Priority Application	PROSECUTION	1
1-24-2005	WCLM	Claims Worksheet (PTO-2022)	PROSECUTION	1
1-24-2005	IMIS	Miscellaneous Internal Document	PROSECUTION	1
1-24-2005	371P	Documents submitted with 371 Applications	PROSECUTION	1
1-24-2005	371P	Documents submitted with 371 Applications	PROSECUTION	1
1-24-2005	371P	Documents submitted with 371 Applications	PROSECUTION	1
1-24-2005	371P	Documents submitted with 371 Applications	PROSECUTION	3
1-24-2005	371P	Documents submitted with 371 Applications	PROSECUTION	26

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